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IS 15030 (2001): Terephthalic Acid [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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भारतीय मानक

टेरीथैलिक ऐसिड — विशिष्टि

Indian Standard

TEREPHTHALIC ACID — SPECIFICATION

ICS 71.080.40

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

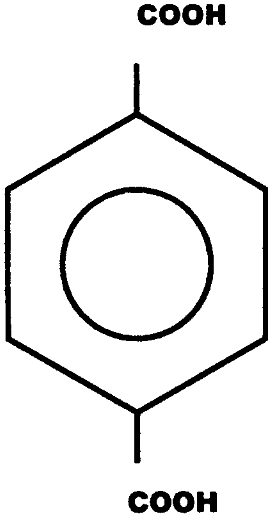
November 2001 **Price Group 7**

Organic Chemicals (Miscellaneous) Sectional Committee, PCD 9

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Terephthalic acid ($C_8H_6O_4$) is derived by oxidation of para-xylene. It is also commercially known as para-phthalic acid; benezene-para-dicarboxylic acid or 1,4 benzenedicarboxylic acid. It is represented by the following structural and molecular formuale:



(Molecular Weight – 176)

[Molecular Formula : $C_6H_4\,(COOH)_2$]

It is used in the production of synthetic resins, fibers and films by combination with glycols.

Composition of the Committee responsible for formulation of this standard is given in Annex K.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*). The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

TEREPHTHALIC ACID — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for pure terephthalic acid.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute the provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below:

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
266 : 1993	Sulphuric acid (*third revision*)
460 (Part 1) : 1985	Specification for test sieves : Part 1 Wire cloth test sieves (*third revision*)
915 : 1975	One-mark volumetric flasks (*first revision*)
1070 : 1992	Specification for water for general laboratory use (*third revision*)
2362 : 1993	Determination of water by Karl Fisher method (*first revision*)
4161 : 1967	Nessler cylinders

3 REQIREMENTS

3.1 Description

The material shall be in the form of white crystalline free flowing powder and free from extraneous impurities like dust and dirt. Terephthalic is insoluble in water, chloroform, ether, acetic acid but slightly soluble in alcohol and completely soluble in alkalis.

3.2 The material shall also comply with the requirements given in Table 1, when tested in accordance to the methods referred in col 4 of the Table 1.

3.3 Particle Size

Not more than 36 percent by mass of the material shall pass through 56 micron IS sieve [*see* IS 460 (Part 1)] and not more than 6 percent by mass of the material shall be retained on 250 micron IS Sieve when tested in accordance to method given in Annex H.

Table 1 Requirements For Terephthalic Acid

(*Clause* 3.2)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test (Ref to Annex) (4)
i)	Moisture content, percent by mass, *Max*	0.2	A
ii)	Colour, in Hazen units, *Max*	10	B
iii)	Ash content, ppm, *Max*	10	C
iv)	Acid value, mg KOH/g	675±2	D
v)	Total of iron, cobalt, manganese and chromium	8(each not greater than 2 ppm)	E
vi)	Total of sodium, magnesium, potassium, calcium and Nickel ppm, *Max*	10 (each not greater than 2 ppm)	F
vii)	4-carboxybenzaldehyde, ppm, *Max*	20	G (Method A, and Method C)
viii)	*p*-toluic acid, ppm, *Max*	150	G (Method A and Method B)

4 PRECAUTIONS IN HANDLING

Terephthalic acid has very low toxicity. The use of dust masks is advisable when handling terephthalic acid. Finely divided organic products which are combustible are liable to explosion should the dust content of the atmosphere be high and if ignition occurs. Good house keeping is, therefore, advisable to minimize the amount of dust when terephthalic acid is handled. The atmosphere of any silo or pneumatic transfer equipment where dust explosions could occur should be blanketed with inert gas to below 10 volume percent oxygen content.

5 PACKING AND MARKING

5.1 The material shall be supplied in bulk containers of 1 000 kg in flexible woven plastic or packages as agreed to between the purchaser and the supplier.

5.2 Marking

The packages shall be marked with the following:

a) Name of the material,
b) Indication of the source of manufacture,
c) Net mass of the material in the container, and
d) Lot or batch number.

5.2.1 *BIS Certification Marking*

The containers may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the rules and regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 TEST METHODS

6.1 Tests shall be conducted according to the method of test referred in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities, which affect the results of analysi.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MOISTURE CONTENT

A-1 GENERAL

Moisture content is determined by using the Karl Fischer method.

A-1.1 Take about 2 g of the material and determine the moisture content by Karl Fischer method as described in IS 2362.

ANNEX B

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF COLOUR — HAZEN

B-1 GENERAL

Two methods, namely, Method A and Method B have been prescribed. In case of dispute, Method A shall be treated as referee method.

B-2 METHOD A

B-2.1 Outline of the Method

The colour of a 5% solution of terephthalic acid in N, N'-Dimethylformamide is compared with that of the colour standard and expressed in terms of Hazen colour units. The Hazen colour which is defined as the colour of an aqueous solution, containing 1 part per million of platinum in the form of chloroplatinic acid and 2 parts per million of cobalt chloride ($COCl_2.6H_2O$).

B-2.2 Apparatus

B-2.2.1 *Nessler Cylinder* — two, 100 ml capacity each (*see* IS 4161).

B-2.2.2 *One Mark Volumetric Flasks* — 250 ml and 500 ml capacities (*see* IS 915).

B-2.2.3 *Erlenmeyer Flask* — 250 ml.

B-2.3 Reagents

B-2.3.1 *Cobaltous Chloride Hexahydrate* — solid.

B-2.3.2 *Concentrated Hydrochloric Acid* — conforming to IS 265.

B-2.3.3 *Chloroplatinic Acid*

Dissolve 250 mg of platinum in small quantity of aqua regia contained in a glass or procelain basis by heating on a water-bath. When the metal has dissolved, evaporate the solution to dryness. Add 1 ml of hydrochloric acid and again evaporate to dryness. Repeat this operation twice.

B-2.3.4 *N,N'-Dimethylformamide*

B-2.4 Preparation of Colour Standard

Dissolve 0.50 g of the cobalt chloride hexahydrate and whole of the chloroplatinic acid (*see* **B-2.3.3**) in 50 ml of the hydrochloric acid. Warm, if necessary, to obtain a clear solution and after cooling, pour into the 500-ml one-mark graduated flask. Dilute with water to the mark. This solution has a colour of 500 Hazen colour units.

B-2.4.1 Pipette 5 ml of the solution (*see* **B-2.4**) into a 250-ml one-mark graduated flask and dilute with water to the mark. The diluted solution corresponds to a colour of 10 Hazen units and should always be freshly prepared.

B-2.5 Procedure

Weigh accurately 5.0 g of the material into a 250-ml Erlenmeyer flask and add 100 ± 0.5ml of N, *N'*-Dimethylformamide. Shake the stoppered flask until all of the sample is dissolved. Transfer the solution into a Nessler cylinder and compare the colour against a white background with the colour standard taken in the other Nessler cylinder. The material shall be taken to have passed the test if the colour of the sample solution is not darker than that of the colour standard.

B-3 METHOD B

B-3.1 General

The method is for the determination of the colour of a solution of pure terephthalic acid in potassium hydroxide.

B-3.2 Outline of the Method

The sample is dissolved in aqueous potassium hydroxide and the colour of the solution measured in Hazen units.

B-3.3 Reagents

Potassium hydroxide solution — 2N aqueous.

B-3.4 Apparatus

B-3.4.1 *Lovibond Nessleriser*

B-3.4.2 *Nessleriser Cylinders* — one matched pair of 50 ml cylinders (*see* IS 4161).

B-3.4.3 *Hazen Discs* — 0.70 and 70-250 Hazen units.

B-3.5 Procedure

Measure 75 ml of 2N potassium hydroxide solution into a 100 ml beaker. With constant stirring carefully add 10.0 g of sample. When dissolved fill a 50 ml Nessleriser cylinder to the mark with the solution and place in the right hand compartment of the Lovibond Nessleriser cylinder to the mark with the solution and place in the right hand compartment of the Lovibond Nessleriser. Fill the second Nessleriser tube to the mark with 2N potassium hydroxide solution and place in the left hand compartment of the Nessleriser. Stand the Nessleriser before a uniform source of white light and compare the colour of the sample with the colours in the standard Hazen disc; rotate the disc until a colour match is obtained.

Report the colour in Hazen units.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF ASH

C-1 APPARATUS

C-1.1 Muffle Furnace

C-1.2 Blast Burner

C-1.3 Platinum Dish — 50 ml.

C-1.4 Micro Balance

C-1.5 Crucible Tongs — made of stainless steel with platinum tips.

C-1.6 Glazed Porcelain Plates

C-2 PROCEDURE

Clean the platinum dish free from grease and salts by soaking in hot chromic acid, washing with 1 : 1 HCl followed by distilled water. Place the dish in the muffle furnace with the help of platinum tipped crucible tongs and heat at 500°C for 30 minutes. Remove the dish from the furnace and place it on a clean porcelain plate which is free of dust particles. Allow the dish to cool for one minute and transfer it to a desiccator which is placed near the micro balance. Take the mass of the empty dish and again with sample of the material to the nearest 0.1 g. Place the dish with material on a triangle supported by a tripod and ignite the sample (*see* Note 1) by means of a suitable burner. After ignition, place the dish in the muffle furnace at 550°C and remove the same from the furnace when all carbon is removed (*see* Note 2). Place the dish with ash in the desiccator for 30 minutes. Take the mass of the dish and its contents.

NOTES

1 A minimum of 300 g of sample is to be incinerated.

2 Once the sample has been reduced to a carbonaceous ash, extreme care must be exercised in handling the dish. If the dish is handled in the usual manner with the tongs, carbon may be transferred to the tips of the tongs and may add carbon to the ash when the dish is removed from the furnace. In order to prevent this source of serious error, turn the tongs with tips pointing upward and pick up the dish by placing the arcs of the tongs around the body of the dish. The dish should not make contact with the refractory lining of the furnace as particles of platinum may be scratched off thus changing its mass.

C-3 CALCULATION

$$\text{Ash, ppm} = \frac{M_3 - M_1}{M_2 - M_1} \times 10^6$$

where

M_1 = mass in g of empty dish,
M_2 = mass in g of empty dish with sample, and
M_3 = mass in g of dish with ash.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ACID VALUE

D-1 GENERAL

The material is dissolved in pyridine, diluted with water and titrated with aqueous sodium hydroxide to phenolphthalein end point.

D-2 REAGENTS

D-2.1 Pyridine

D-2.2 Phenolphthalein— 1 percent solution in ethanol.

D-2.3 Sodium Hydroxide — 0.5 M aqueous solution.

D-3 PROCEDURE

Weigh accurately about 1 g of the material into a 100-ml beaker. Add 25 ml of pyridine and stir until all the sample is dissolved. Add 25 ml of water and two drops of phenolphthalein indicator. Titrate with 0.5 M sodium hydroxide solution to a permanent pink end point. Carry out a blank determination simultaneously excluding the material.

D-4 CALCULATION

$$\text{Acid value, mg KOH/g} = \frac{(V_1 - V_2) \times M \times 56.1}{W}$$

where

V_1 = Volume in ml of sodium hydroxide consumed with sample,

V_2 = Volume in ml of sodium hydroxide consumed with blank,

M = Molarity of sodium hydroxide, and

W = Mass of sample taken for the test.

ANNEX E

[*Table* 1, *Sl No.*(v)]

DETERMINATION OF IRON, COBALT, MANGANESE AND CHROMIUM

E-1 PRINCIPLE

The material is ashed in two separate dishes in presence of sulphuric acid. Ash in one dish is dissolved in concentrated hydrochloric acid and cobalt and manganese are determined by atomic absorption spectrophotometric method. Ash in the other dish is fused with potassium hydrogen sulphate, dissolved in concentrated hydrochloric acid and iron and chromium are determined by atomic absorption spectrophotometric method. In both cases air/acetylene flame is used.

E-2 APPARATUS

E-2.1 Atomic Absorption Spectrophotometer (AAS) — For determination of these elements using AAS, details of test methods as given in relevant Indian Standard (*see* Annex J) may be referred. Typical working conditions of AAS are given below for guidance only:

	Iron	*Cobalt*	*Manganese*	*Chromium*
a) Wave-length, nm	248.3	240.7	279.5	357.9
b) HV	700	620	620	460
c) Band pass	0.3	0.3	0.3	0.3
d) Air	16	16	16	16
e) Fuel (acetylene)	6	6	6	6
f) Burner height, mm	8	7	5	8
g) Lamp current, mA	7	7	3	5
h) Scale expansion	1	2	1	2
j) Burner Angle	0	0	0	0

E-3 REAGENTS

E-3.1 Concentrated Hydrochloric Acid— *see* IS 265.

E-3.2 Concentrated Sulphuric Acid— *see* IS 266.

E-3.3 Potassium Hydrogen Sulphate — fine powder.

E-3.4 Stock Iron Solution — 1 000 ppm (*m/v*).

E-3.5 Stock Cobalt Solution—1 000 ppm (*m/v*).

E-3.6 Stock Manganese Solution— 1 000 ppm (*m/v*).

E-3.7 Stock Chromium Solution —1 000 ppm (*m/v*).

E-4 PREPARATION OF STANDARD SOLUTIONS

E-4.1 Standard Working Solutions

Pipette 10.0 ml of each of the stock solutions (*see* **E-3.4** to **E-3.7**) into a 1 000-ml flask and make up to the mark to given 10 ppm (*m/v*) standard working solutions. These shall be prepared fresh everytime before testing.

E-4.1.1 *Standard Iron and Chromium Solutions*

Take 5,10 and 20 ml of both standard working solutions of iron and chromium of 10 ppm (*m/v*) (*see* **E-4.1**) in each of four 100-ml volumetric flasks. Add 1 g of ground potassiun hydrogen sulphate and 5 percent hydrochloric acid and shake to dissolve. Dilute to the mark with 5 percent HCl to give standard solutions containing 0.5, 1.0, 1.5 and 2.0 ppm (*m/v*) of iron and chromium.

E-4.1.2 *Standard Cobalt and Manganese Solutions*

Take 5, 10, 15 and 20 ml of both standard working solutions of cobalt and manganese of 10 ppm (*m/v*) (*see* **E-4.1**) in each of four 100-ml volumetric flasks. Make up to the mark with 5 percent HCl and shake well. This gives standard solutions containing 0.5, 1.0, 1.5 and 2.0 ppm (*m/v*) of cobalt and manganese.

E-5 PROCEDURE

Weigh accurately about 25 g of sample into two separate silica dishes. Wet thoroughly with 5 ml of concentrated sulphuric acid and char completely under a vertical heater. Place the dishes in the entrance of a muffle furnace with the door open, and fume until most of the carbon has been removed. Move the dishes further into the muffle furnace, close the door gently and leave until all the carbon has been removed. Care shall be taken that the sample does not ignite.

E-5.1 Add 5 ml of concentrated hydrochloric acid to one of the dishes, warm, if necessary, to dissolve and transfer quantitatively to a 100-ml volumetric flask. Make up to the mark with water. Run through in following order; respective standards; samples and standards again for cobalt and manganese using given conditions (*see* **E-2.1**).

E-5.2 Fuse the residue from the other dish with 1.00 g of ground potassium hydrogen sulphate ($KHSO_4$) and a few drops of concentrated sulphuric acid over a Bunsen burner. Cool, add 5 ml of concentrated hydrochloric acid, warm, if necessary to dissolve and transfer quantitatively to a 100 ml volumetric flask and make up to the mark with water. Prepare a blank by taking 1.0 g of ground potassium hydrogen sulphate $KHSO_4$ and a few drops of concentrated sulphuric acid. Run through in following order; respective standards; blank samples and standards again for iron and chromium, using the given conditions (*see* **E-2.1**).

E-5.3 Calculation

The respective metals

(iron, chromium, cobalt, manganese), ppm = $\frac{C_1 \times 100}{M}$

where

C_1 = iron, cobalt, chromium, manganese content in solution derived from sample, ppm and

M = mass of the sample taken for test, g.

ANNEX F

[*Table* 1, *Sl No.*(vi)]

DETERMINATION OF SODIUM, MAGNESIUM, POTASSIUM, CALCIUM AND NICKEL

F-1 PRINCIPLE

The sample is ashed in the presence of sulphuric acid. The ash is dissolved in concentrated hydrochloric acid. Concentration of sodium, potassium, and nickel are determined by atomic absorption spectrophotometry in an air/acetylene flame. Concentration of calcium and magnesium are determined by atomic absorption spectrophotometry in a nitrous oxide/acetylene flame.

F-2 APPARATUS

F-2.1 Atomic Absorption Spectrophotometer — For determination of these elements using AAS, details of test methods as given in relevant Indian Standard (*see* Annex J) may be referred. Typical conditions only for guidance are given below. The wavelengths given are the true atomic line wavelengths and may not correspond exactly to the wavelength dial of the instrument:

	Sodium	*Nickel*	*Calcium*	*Magnesium*	*Potassium*
a) Wave length, nm	589.6	232.0	422.7	285.2	766.5
b) Band pass, nm	0.5	0.15	0.5	0.5	2
c) Burner Height, nm	7	7	7	7	7
d) Oxidant Air	Air	N_2O	N_2O	N_2O	N_2O
e) Lamp current, mA	4	7	5	4	*
f) HV	530	700	530	460	800
g) Integration time, sec	1	1	1	1	1

*emission mode.

F-3 REAGENTS

F-3.1 Hydrochloric Acid — *see* IS 265.

F-3.2 Sulphuric Acid — IS 266.

F-3.3 Stock Sodium Solution — 1 000 ppm (*m/v*).

F-3.4 Stock Potassium Solution — 1 000 ppm (*m/v*).

F-3.5 Stock Calcium Solution — 1 000 ppm (*m/v*).

F-3.6 Stock Magnesium Solution — 1 000 ppm (*m/v*).

F-3.7 Stock Nickel Solution — 1 000 ppm (*m/v*).

F-4 PREPARATION OF STANDARD SOLUTIONS

F-4.1 Standard Working Solutions

Pipette 10 ml of each of the stock solutions (*see* **F-3.3** to **F-3.7**) into 1 000-ml flask and dilute each to the mark to give 10 ppm (*m/v*) of standard working solution. These shall be prepared fresh every day before testing.

F-4.2 Standard Sodium, Potassium, Calcium, Magnesium and Nickel Solutions

Take 10 ml concentrated hydrochloric acid into each of the five 100-ml volumetric flasks. Add by means of pipette 2, 5, 10, 20 and 50 ml of each of the standard working solutions (*see* **F-4.1**) into each of the volumetric flasks and dilute to the mark with water to give standard solutions of 0.2, 0.5, 1.0, 2.0 and 5.0 ppm (*m/v*) of each of the elements. Prepare a zero standard solution by pipetting 10 ml of concentrated hydrochloric acid into a 100-ml volumetric flask and diluting to the mark with demineralized water.

F-5 PROCEDURE

Weigh accurately about 25 g of sample into a silica dish. Wet thoroughly with 5 ml of concentrated sulphuric acid and char completely on a temperature controlled heater. Prepare a blank by adding 5 ml of concentrated sulphuric acid to a silica dish and evaporate on a temperature controlled heater. Place the dishes into a muffle furnace at 550°C, close the door and leave until all the carbon has been removed. Care shall be taken that the sample does not ignite. Remove the dishes, cover with a watch glass and cool. Add 2.5 ml of concentrated hydrochloric acid to each dish, cover with a watch glass and place each dish ona hot plate to simmer for 5 minutes. Cool and dilute the sample with 10 ml of demineralized water. Transfer the sample and blank to two 25 ml volumetric flasks and dilute to the mark with water.

F-5.1 Using the absorption mode of the instrument determine sodium, calcium, magnesium, and nickel on the sample and blank against the prepared standards.

F-5.2 Using the emission mode of the instrument determine potassium on the sample and blank.

F-6 CALCULATION

$$\text{Respective metal content, ppm} = \frac{25\,(C_1 - C_2)}{W}$$

where

C_1 = concentration of Na, Mg, Ni and K in sample solution, ppm;

C_2 = concentration of Na, Ca, Mg, Ni and K in blank, ppm; and

W = mass of sample taken for test g.

ANNEX G

[*Table* 1, *Sl No.*(vii) *and* (viii)]

DETERMINATION OF 4 - CARBOXYBENZALDEHYDE (4-CBA) AND *p*–TOLUIC ACID (*p*-TA)

G-1 GENERAL

Three methods namely Method A, Method B and Method C have been prescribed. Method A and Method C may be used for determination of 4-carboxybenzaldehyde and Method A and Method B may be used for determination of *p*-toluic acid. Method A is HPLC method, Method B is a GC method and Method C is polarographic method.

G-2 METHOD A : DETERMINATION OF 4-CARBOXYBENZALDEHYDE AND *p*-TOLUIC ACID (*p*-TA) BY HPLC METHOD

G-2.1 Principle

4-Carboxybenzaldehyde and *p*-toluic acid in terephthalic acid is determined by high performance liquid chromatography (HPLC) using ammonium-phosphate as buffer solution/mobile phase and a 254 nm absorbance detector.

G-2.2 Apparatus

G-2.2.1 *A Suitable HPLC System*

G-2.2.2 *p*H *Meter*

G-2.2.3 *Solvent Delivery System*

G-2.2.4 *Absorbance Detector*

G-2.2.5 *Syringe Loading Injector* — with a 20 µl sample loop.

G-2.2.6 *Data Processor, Computing Integrator*

G-2.2.7 *Syringe* — 100 µl capacity.

G-2.2.8 *Beakers* — 100 ml.

G-2.3 Reagents

G-2.3.1 *Ammonium Phosphate* — 0.25 M. Dissolve 28.75 g of ammonium phosphate (monobasic) in 1 000 ml of water.

G-2.3.2 *Acetonitrile*

G-2.3.3 *Ammonium Hydroxide* — 3.7 percent.

G-2.3.4 *Benzyl Alcohol*

G-2.3.5 *Phosphoric Acid*

G-2.4 Preparation of Buffer Solution/Mobile Phase

Weigh accurately 28.75 g of ammonium phosphate in 1 000-ml volumetric flask and dissolve in about 900 ml of demineralized water. Pipette 30 ml of acetonitrile into the flask and dilute to the mark (*see* Note 1). Place the solution in a 1 000-ml beaker and adjust the *p*H to 4.25 with concentrated NH_4OH, if the *p*H is to be raised or concentrated H_3PO_4 if the *p*H is to be lowered, *p*H meter shall be calibrated with a 4 *p*H buffer solution (*see* Note 2). Vacuum filter the solution through a 0.22 m millipore filter or 0.6 m millipore filter. Stir the solution under reduced pressure to remove dissolved gas.

NOTES

1 The concentration of ammonium phosphate and acetonitrile may be required to be changed as the column gets older to maintain the terephathalic acid/ *p*-toluic acid separation. Lower concentration of acetonitrile [2 per cent to 4 percent (*v/v*) will normally broaden peak width and move the peaks for *p*-toluic acid and benzoic acid toward the large terephthalic peak. Higher concentration of acetonitrile 4.5 to 6.5 percent (*v/v*)] will narrow peak width and move the *p*-toluic acid benzoic acid away from the terephthalaic acid peak. However, there will be a tendency for benzoic acid to move into terephthalic acid peak. Raising concentration of acetonitrile is common as a column gets older and peak separation is less marked.

2 Changes in the lower *p*H range will increase peak separation and broaden peak width, and changes in the higher *p*H range will reduce retention time which will cause superimposition of peaks upon adjacent peaks. The highest permissible *p*H shall be 4.5.

G-2.5 Preparation of Column

Pack the column with hexane and then flush with 250 ml of acetonitrile to dissolve hexane. Flush the column again for 15 minutes with water at a flow rate of 1.3 ml per minute. Run the buffer solution (*see* **G-2.4**) through the column for 16 hours at a rate of 1.3 ml per minute to stabilize the column.

G-2.6 Procedure

Weigh accurately 0.2 ± 0.000 5 g of sample into a 100 ml beaker and dissolve with 20 ml of ammonium hydroxide solution (*see* **G-2.3.3**). Add 20 ml of water followed by 10 µl of benzyl alcohol. Adjust the *p*H of the solution to 6.8 to 7.2 with concentrated phosphoric acid (*see* **G-2.3.5**). Inject 100 µl of sample solution into injection valve with valve in load position. Turn the valve to inject position and press start button on computing integrator interface. Inject two syringes (100 µl) of water into injection valve with it in the inject position and press start button on computing integrator interface. Inject two syringes (100 µl) of water into injection valve with it in the inject position.

G-2.6.1 The instruments should be calibrated by analysis of one of two samples which have been previously analysed by polarography. The sample with known quantity of 4 CBA may be tested for calibration of equipment.

G-2.7 Calculation

Calculate and report the contents of 4-carboxybenzaldehyde and *o*-toluic acid by means of computing integrator.

G-3 METHOD B: DETERMINATION OF PARA-TOLUIC ACID (p-TA) IN PURE TEREPHTHALIC ACID BY GAS CHROMATOGRAPHIC METHOD

G-3.1 General

This method is used for the determination of para toluic acid in terephthalic acid in the range of 0-200 ppm.

G-3.2 Summary of the Method

The sample is dissolved in tetramethyl ammonium hydroxide solution and injected into an injector (pyrolysis unit). The resulting esters are then separated by gas chromatography using a heart cut system for estimation of the para toluic acid content.

G-3.3 Principle

PTA dissolved in tetramethyl ammonium hydroxide (TMA) is injected in pyrolysis block maintained 400 ± 5 °C. All the acids get converted into respective esters. Thus, para toluic acid gets converted to methyl para toluate (MPT).

This methyl *p*-toluate (MPT) is determined by separation technique on GC column using heart cut system.

G-3.4 Instrument

G-3.4.1 *Gas Chromatograph*

Any suitable gas chromatograph with flame ionization detector equipped with heart-cutting system with a pyrolysis unit heated to 400 °C. Integrator – A suitable computing integrator and syringe – micro- liter syringe of 10 µl size.

G-3.4.1.1 *Chromatographic column*

a) Pre-column : 1/8" Stainless steel 1 meter column packed with 10% SP-1000 on 80-100 mesh AW Chromasorb W.
b) Main-column : 1/8" Stainless steel 2 meter column packed with 10% SP-1000 on 80-100 mesh AW Chromasorb W.

G-3.4.1.2 *Column conditioning*

Condition the column before use for 12 h having nitrogen carrier flow of 30 ml/min.

G-3.4.1.3 *Reagents and consumables*

a) Instrument air : Pure with very low hydrocarbons
b) Hydrogen : Commercial grade
c) Nitrogen : Commercial grade
d) Para toluic acid (p-TA) : 99.0% purity
e) Tetramethyl ammonium hydroxide : 25% aquous solution
f) Terephthalic acid : Pure containing known amount of para-toluic acid (p-TA)

NOTE — Tetramethyl ammonium hydroxide is corrosive and causes burns to the skin.

G-3.5 Method for Establishing GC for p-TA Analysis

G-3.5.1 Set the condition as follows:

Injector pyrolysis block	= 400 °C
Detector temperature	= 200 °C
Column/oven (isothermal) temperature	= 180 °C
Carrier gas	= Nitrogen

G-3.5.2 Energize the solonoid valve manually via push buttons on flow switching unit (FSU) and set the primary nitrogen regulator (stream 1) to the 27 psig (1.9 Kg/cm^2). At this time secondary nitrogen regulator must be turned off and heart cut needle valve (in FSU) should be fully open.

G-3.5.3 Allow the system to attain equilibrium that may take about 15-20 minutes, and note the natural mid point pressure (secondary nitrogen/stream 2 pressure gauge).

G-3.5.4 Measure the carrier gas flow rate at FID vent (Air/H_2 off). It should be 5 ml in 14.0 + 0.1 sec by bubble flow meter. If it is not so, adjust the flow using PF (premium regulator 1).

NOTE—To achieve the above mentioned rate, slight variation in pressure may be necessary, if pressure required to check for column leaks, septum leak, etc, is higher.

G-3.5.5 De-energize the solonoid valve manually by push bottom on FSU. Now the mid-point pressure will decrease gradually via the heart cut vent.

G-3.5.6 Using secondary nitrogen pressure regulator (PR2), increase the mid-point pressure to 0.5 psig greater than the noted natural mid-point pressure.

G-3.5.7 With the solonoid valve energized, measure the flow rate at the FID vent (Air/H_2off). Note this value.

G-3.5.8 De-energize the solonoid valve and adjust the heart cut needle valve to give a flow rate equal to measured at FID vent, that is 5 ml in 12.0 min.

G-3.5.9 Set the FID air and hydrogen to the flow rates below:

Hydrogen pressure = 10.5 psig (0.75 Kg/cm^2) = 31.3 ml/min + 5.0
Air pressure = 6.5 psig (0.45 Kg/cm^2) = 258.6 ml/min + 10.0

G-3.5.10 Light the flame and allow the system to stabilize overnight.

G-3.6 Calibration

G-3.6.1 *Three Point Calibration*

G-3.6.2 *Preparation of Standards*

a) Weigh accurately 0.50 g of para toluic acid, dissolve in methanol and dilute to 100 ml in a volumetric flask, with methanol. Take 1.0 ml of this solution and dilute to 100 ml with distilled water.

b) Weigh accurately 0.25 g of para toluic acid, dissolve in methanol and dilute to 50 ml in a volumetric flask, with methanol. Take 1.0 ml of this solution and dilute to 100 ml with distilled water.

c) Take 5 vials numbered 1-5 and weigh 1.00 g of TA of low p-TA content (<100 ppm) into each vial. Then add solutions A and B, water and tetramethyl ammonium hydroxide (25%) as detailed in the table below:

Vial	1	2	3	4	5
TA gm	1.00	1.00	1.00	1.00	1.00
p-TA solution A ml	NIL	1.0	NIL	3.0	NIL
p-TA solution B ml	NIL	NIL	2.0	NIL	4.0
H20 ml	5.0	4.0	3.0	2.0	1.0
TMA ml	5.0	5.0	5.0	5.0	5.0
ppm p-TA Added	0	50	100	150	200

Plot a calibration graph of peak area (corrected for blank *Versus* p-TA added for preparing the standards.

Calculate the response factor for each standard using following formula:

$$\text{Response factor} = \frac{\text{Peak area}}{\text{p - TA ppm of standard solution}}$$

G-3.7 Preparation of Sample

Weigh 1.0 ± 0.1 g paratoluic acid (p-TA) sample in paper boat. Transfer the sample in vial, add 100 ml 1:1 tetra methyl ammonium hydroxide (TMA) water solution and dissolve the sample.

Inject 5.0 μl of sample solution in GC using 10 μl hamilton syringe.

G-3.8 Calculation

After getting the correct retention time of peak of interest and calculated response factor of standard, feed it in computing integrator.

At the end of run, integrator will automatically gives the ppm of para-toluic acid present in sample *OR* we can calculate as:

$$\text{ppm p-TA Acid} = \frac{\text{Peak area of sample} \times \text{standard concentation}}{\text{Peak area of standard}}$$

G-4 METHOD C DETERMINATION OF 4 CARBOXYBENZALDEHYDE IN PURE TEREPHTHALIC ACID USING POLORAGRAPHIC ANALYZER

G-4.1 General

This method is for the determination of 4 carboxy benzaldehyde (4 CBA) in pure terephthalic acid in the range 0 to 50 ppm.

G-4.2 Summary of the Method

The sample is dissolved in 1 M potassium hydroxide solution and 0.01 % of polyethylene glycol (PEG 6000) is added. After deoxygenating with nitrogen the differential pulse polarogram is recorded and the height of the carbonyl reduction wave at –1.32 V (relative to a saturated calomel electrode) is measured. This peak height is proportional to the concentration of 4CBA in the sample and from a calibration graph, constructed using standard solutions, the concentrations of 4CBA is determined.

G-4.3 Principle

Polarographic technique is used for those components in solution that can be electrochemically oxidized or reduced. In this potential (voltage) is applied to sample via a conductive electrode that is dropping mercury electrode which is also called as working electrode. Potential is scanned over region of interest, much as the wavelength of light is scanned in spectroscopic measurements. When component is reduced/oxidized at particular potential current will flow at working electrode and the potential at which the current is one half the diffusion value is half wave potential E ½. This E ½ is characteristic of particular substance undergoing reaction and is useful in identifying the solution constituents. Table of E ½ values for various organic and inorganic species is available in reference books.

For determination of 4CBA, differential pulse polarographic technique is used in which voltage is applied to the cell in pulses. Current produced after reduction of 4 CBA is measured twice for each drop, first just before pulse is applied and second just before pulse ends. The difference in two readings is signal which is fed to X-Y flat bed recorder and peak proportional to concentration of 4CBA is recorded.

G-4.4 Interference

a) Dissolved oxygen is expelled from the test solution with inert gas such as argon or nitrogen or otherwise polarogram of dissolve oxygen will interfere with the sample.

b) Polyethylene glycol 6000 is added in test solution to suppress the polarographic maximum which normally interferes in peak of interest.

G-4.5 Instruments

A suitable polarographic analyzer fitted with dropping mercury electrode and XY flat bed recorder.

G-4.6 Reagents and Consumables

1M Potassium Hydroxide solution : Dissolve 330 gm of AR Potassium hydroxide in water and make up to 5 litre.

Pure TA : Containing < 5 ppm 4 CBA is suitable.

1%m/v PEG 6000 : Dissolve 1.0 gm of PEG 6000 GR grade in water and make upto 100 ml in standard flask shake well to ensure complete solution.

4 Carboxy Benzaldehyde : AR Grade. Purity – 99%.

G-4.7 Calibration Standard

Weigh 0.100 0 gm of pure 4 CBA into 50 ml beaker. Add approximately 30 ml of 1M potassium hydroxide and stir to dissolve. Quantitatively transfer this solution to a100 ml volumetric flask; and dilute to mark, with 1M KOH. Call this solution '*A*'.

1 ml solution '*A*' = 1 mg 4CBA.

Pipette 10 ml of solution *A* into 100 ml volumetric flask and dilute to the mark with 1M potassium hydroxide. Call this solution, *B*G.

1ml solution *B* = 100 µgm 4CBA.

NOTES

1 Solution *A* and *B* are not stable and should be made as and when required.

2 If 4CBA is dissolved in warm potassium hydroxide solution, yellow colour can occur, indicative of chemical reaction. If this occurs, 4CBA solution should be discarded and cold potassium hydroxide solution should be used before making standard.

3 Argon/Nitrogen – approximately 1.5 psi for 16 minutes to ensure absence of oxygen peak.

4 Mercury : Polarographic Grade or AR Grade.

CAUTION — As mercury is very poisonous and its vapours are harmful, mercury should be handled very carefully. If any spillage occurs, mercury should be removed immediately using vacuum.

G-4.8 Calibration

Three point calibration:

a) Into six 100 ml volumetric flasks weigh 5.00 gm of pure TA.

b) Pipette 1ml of 1% m/v PEG 6000 solution into each flask and add approximately 75ml of 1M potassium hydroxide. Stopper the flasks and shake vigorously; to dissolve TA.

c) To series of 100 ml flasks add from pipette 0, 0.5, 1.0, 1.5, 2.0, 2.5 ml of solution B respectively.

d) Make upto the mark with 1M KOH. Shake well to mix and then record the polarogram for each standard as described in procedure.

e) Plot a calibration curve of µgm 4CBA *v/s* peak height (corrected for blank).

f) Calculate the mean factor as F = 1/Slope.

G-4.9 Procedure

G-4.9.1 Conditions

The following operating conditions for polarographic analyser are given for guidance only.

I *Polarographic Analyser*

Initial potential	:	–0.90 V
Final potential	:	–1.40 V
Scan rate	:	–2mv/sec
Mode	:	Diff.pulse
Pulse height	:	50MV
Current range	:	10µA
Equilibrium time	:	15 sec.
Drop time	:	1 sec.
Replication cycles	:	1
Scan rate multiplier	:	X1
Filter (DC only)	:	OFF

II *Dropping Mercury Electrode*

Drop Size	:	Large (out of large, medium, small)
Mode	:	DME/HMDE (Out of SMDE, DME, HMDE)

III *XY Flat Bed Recorder*

Var/Cal 'X'	:	Cal 0.1 mv/cm
Var/Cal 'Y'	:	Cal 01 mv/cm

G-4.9.2 Sample Preparation

a) Transfer quantitatively 5.0 ± 0.01 gm of PTA into 100 ml volumetric flask.

b) Pipette 1 ml of 1% w/v PEG 6000 solution to the flask.

c) Add 75 ml of 1M KOH and stopper the flask. Shake vigorously till all TA is dissolved.

d) Make upto the mark with 1M KOH and mix well.

e) Pour approximately 12ml of this solution into polarographic cell and deoxygenate with nitrogen for 16 minutes with slow purging.

f) Check all the operating conditions as given.

g) Check that dropping mercury electrode, calomel electrode and platinum electrode are well immersed in the solution.

h) Start the polarograph by pressing 'RUN' and start the recorder and lower the recorder pen.

j) On completion of plarogram, swith 'OFF' the recorder.
k) Remove the cell. Clean the cell, capillary and electrodes with distill water and refill the cell with distilled water and put it back.
m) Measure the peak height in mm.

G-4.10 Calculation

$$\text{4-CBA ppm } m/m = \frac{\text{Height in mm} \times F}{\text{Mass of sample}}$$

where

F = Factor from calibration curve.

ANNEX H

(*Clause* 3.3)

PARTICLE SIZE DISTRIBUTION OF PURE TEREPHTHALIC ACID (SIEVE ANALYSIS)

H-1 GENERAL

This method is for the determination of particle size distribution of terephthalic acid using standard test sieves in the range of 56 to 250 micron.

H-2 SUMMARY OF THE METHOD

The sample with addition of little carbon is sieved using standard sieves and sieve shaker from a known period of time. The retained material on each sieve is weighed by difference and particle size distribution is determined.

H-3 PRINCIPLE

Particle size distribution is determined by using technique, 'Test sieving' with the held of vertically vibrating throw-principle sieve shaker.

Sieve shaker contains an electronically controlled electromagnet mounted on sturdy cast base which vibrates. The vibrating plate is connected to base by springs. 3 000 vibrations per minute are transmitted to the plate, which enable the sieving. To reduce the sieving time and to remove static current activated carbon is added to the sample as a sieving aid.

H-4 EQUIPMENT

a) Sieve shaker; and
b) Set of 200 mm diameter stainless steel sieves of 250, 160, 125, 100, 80,63,56 microns respectively with lid and receiver.

H-5 REAGENTS

Activated carbon (LR Grade).

H-6 PROCEDURE

Weigh accurately 50 ± 0.1 gm PTA sample and 0.25 gm of activated carbon. Mix gently, place the sieve set arranged in descending order that is from 250 to 56 mm on the platform. Transfer the carbon mixed sample onto the top sieve. Tighten the sieve stack with the help of belt. Set the amplitude control switch on. Set mode – Inter and keep interval. Allow to oscillate for 10 minutes by setting timer. Weigh portion retained on each sieve by difference that is by taring the sieve having material retained and weighing after subsequent cleaning with vacuum cleaner. Record the mass in g.

H-7 Calculation

$$\text{Percent m/m retained on each sieve} = \frac{A \times 100}{W}$$

where

A = Mass of material retained in g, and
W = Mass of original sample.

Record the percent retained and cumulative on each sieve individually. Plot the graph of cumulative percent against sieve size in microns on semi-logarithmic graph paper. Read out the microns value at level of 50 percent cumulative. Record it as a mean particle size of TA sample.

ANNEX J

(*Clauses* E-2.1 *and* F-2.1)

LIST OF INDIAN STANDARDS FOR DETERMINATION OF IMPURITIES USING ATOMIC ABSORPTION SPECTROPHOTOMETRY

IS No.	*Title*
10614 : 1983	Method for atomic absorption spectrophotometric determination of sodium and potassium
12046 : 1987	Method for determination of manganese by atomic absorption spectrophotometry
12074 : 1987	Method for determination of lead by atomic absorption spectrophotometry
12122 : 1987	Method for determination of nickel by atomic absorption spectrophotometry
12491 : 1988	Method for determination of

IS No.	*Title*
	magnesium by atomic absorption spectrophotometry
13319 : 1992	Determination of chromium by atomic absorption spectrophotometry — Test method
13320:1992	Determination of iron by atomic absorption spectrophotometry — Test method

ANNEX K

(*Foreword*)

COMMITTEE COMPOSITION

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Organization	*Representative(s)*
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